SVENSK STANDARD
SS-EN 10351:2011

Fastställd/Approved: 2011-03-08
Publicerad/Published: 2011-03-30
Utgåva/Edition: 1
Språk/Language: engelska/English
ICS: 77.040.30; 77.140.10; 77.140.45

SWEDISH STANDARDS INSTITUTE

Kemisk analys av järn och stål – Analys av olegerat och låglegerat stål med optisk emissionsspektrometri med induktivt kopplat plasma – Bestämning av Mn, P, Cu, Ni, Cr, Mo, V, Co, Al (total) och Sn [Rutinmetod]

Chemical analysis of ferrous materials – Inductively coupled plasma optical emission spectrometric analysis of unalloyed and low alloyed steels – Determination of Mn, P, Cu, Ni, Cr, Mo, V, Co, Al (total) and Sn [Routine method]

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The European Standard EN 10351:2011 has the status of a Swedish Standard. This document contains the official version of EN 10351:2011.

Upplysningar om sakinnehållet i standarden lämnas av SIS, Swedish Standards Institute, telefon 08-555 520 00. Standarder kan beställas hos SIS Förlag AB som även lämnar allmänna upplysningar om svensk och utländsk standard.

Information about the content of the standard is available from the Swedish Standards Institute (SIS), telephone +46 8 555 520 00. Standards may be ordered from SIS Förlag AB, who can also provide general information about Swedish and foreign standards.

Denna standard är framtagen av kommittén för Kemiska analysmetoder för metaller, SIS/TK 122.

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EUROPEAN STANDARD

NORME EUROPÉENNE

EUROPÄISCHE NORM

EN 10351

March 2011

ICS 77.140.10; 77.140.45

English Version

Chemical analysis of ferrous materials - Inductively coupled plasma optical emission spectrometric analysis of unalloyed and low alloyed steels - Determination of Mn, P, Cu, Ni, Cr, Mo, V, Co, Al (total) and Sn [Routine method]

Analyse chimique des matériaux ferreux - Analyse des aciers non alliés et faiblement alliés par spectrométrie d'émission optique avec source à plasma induit - Détermination de Mn, P, Cu, Ni, Cr, Mo, V, Co, Al (total) et Sn [Méthode de routine]

Chemische Analyse von Eisenwerkstoffen - Analyse von unlegierten und niedrig legierten Stählen mittels optischer Emissionsspektrometrie mit induktiv gekoppeltem Plasma - Bestimmung von Mn, P, Cu, Ni, Cr, Mo, V, Co, Al (gesamt) und Sn [Routineverfahren]

This European Standard was approved by CEN on 15 January 2011.

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Management Centre: Avenue Marnix 17, B-1000 Brussels

Ref. No. EN 10351:2011: E

Contents

Page

Foreword

This document (EN 10351:2011) has been prepared by Technical Committee ECISS/TC 102 “Methods of chemical analysis for iron and steel”, the secretariat of which is held by SIS.

This European Standard shall be given the status of a national standard, either by publication of an identical text or by endorsement, at the latest by September 2011, and conflicting national standards shall be withdrawn at the latest by September 2011.

Attention is drawn to the possibility that some of the elements of this document may be the subject of patent rights. CEN [and/or CENELEC] shall not be held responsible for identifying any or all such patent rights.

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1 Scope

This European Standard specifies an inductively coupled plasma optical emission spectrometry routine method for the analysis of unalloyed and low alloyed steels, whose iron content shall be at least 95 %.

This method is applicable to the elements listed in Table 1 within the ranges shown.

Table 1 — Application ranges

Element	Mass fraction %	
	min.	max.
Mn	0,005	2,00
P	0,005	0,05
Cu	0,005	0,80
Ni	0,010	2,00
Cr	0,010	1,60
Mo	0,005	0,80
V	0,002	0,40
Co	0,002	0,10
Al (total)	0,020	0,30
Sn	0,001	0,10

In all cases, the ranges specified can be extended or adapted (after validation) for the determination of other mass fractions, provided that the iron content in the samples under concern is above 95 %.

Other elements may be included. However such elements and their mass fractions should be carefully checked, taking into account the possible interferences, the sensitivity, the resolution and the linearity criteria of each instrument and each wavelength.

Depending also on the sensitivity of each instrument, suitable dilutions of the calibration and the test sample solutions may be necessary.

Moreover, even if the method described is "multi elemental", it is not absolutely necessary to carry out the determination of all the elements of its scope simultaneously: the measurement conditions have to be optimised by each laboratory, depending on the performances of each apparatus available.

NOTE 1 The accuracy of the method is unsatisfactory for phosphorus contents from 0,05 to 0,1 %.

NOTE 2 The trueness of the method couldn't be checked for vanadium contents below 0,05 %.

NOTE 3 The precision of the method is unsatisfactory for aluminium (total) contents below 0,02 %.

2 Normative references

The following referenced documents are indispensable for the application of this document. For dated references, only the edition cited applies. For undated references, the latest edition of the referenced document (including any amendments) applies.

CEN/TR 10345:2008, *Guideline for statistical data treatment of inter laboratory tests for validation of analytical methods*

EN ISO 14284, *Steel and iron — Sampling and preparation of samples for the determination of chemical composition (ISO 14284:1996)*

ISO 648, *Laboratory glassware — One-mark pipettes*

ISO 1042, *Laboratory glassware — One-mark volumetric flasks*

ISO 5725-1:1994, *Accuracy (trueness and precision) of measurement methods and results — Part 1: General principles and definitions*

ISO 5725-2:1994, *Accuracy (trueness and precision) of measurement methods and results — Part 2: Basic method for the determination of repeatability and reproducibility of a standard measurement method*

ISO 5725-3:1994, *Accuracy (trueness and precision) of measurement methods and results — Part 3: Intermediate measures of the precision of a standard measurement method*

3 Principle

Dissolution of a test portion with nitric and hydrochloric acids. Filtration and ignition of the acid insoluble residue. Removal of silica with hydrofluoric acid. Fusion of the residue with a mixture of orthoboric acid and potassium carbonate, dissolution of the melt with acid and addition of this solution to the reserved filtrate.

After suitable dilution and, if necessary, addition of an internal reference element, nebulisation of the solution into an inductively coupled plasma optical emission spectrometer and measurement of the intensity of the emitted light from each element (including, where relevant, the intensity of the internal reference element).

4 Reagents

During the analysis, use only reagents of recognised analytical grade and only distilled water or water of equivalent purity.

The same reagents should be used for the preparation of calibration solutions and of sample solutions.

4.1 Hydrochloric acid, HCl (ρ_{20} = 1,19 g/ml)

4.2 Hydrochloric acid, solution 1 + 1

Add 500 ml of hydrochloric acid (4.1) to 500 ml of water.

4.3 Nitric acid, HNO_3 (ρ_{20} = 1,33 g/ml)

4.4 Nitric acid, solution 1 + 1

Add 500 ml of nitric acid (4.3) to 500 ml of water.

4.5 Hydrofluoric acid, HF (ρ_{20} = 1,13 g/ml)

WARNING — Hydrofluoric acid is extremely irritating and corrosive to skin and mucous membranes producing severe skin burns which are slow to heal. In the case of contact with skin, wash well with water, apply a topical gel containing 2,5 % (mass fraction) calcium gluconate, and seek immediate medical treatment.

4.6 Sulphuric acid, H_2SO_4 (ρ_{20} = 1,84 g/ml)

4.7 Sulphuric acid, solution 1 + 1

Add 25 ml of sulphuric acid (4.6) to 25 ml of water and allow it to cool.

4.8 Fusion reagent

4.8.1 Fusion mixture

Mix one part by mass of orthoboric acid, H_3BO_3 and one part of potassium carbonate anhydrous, K_2CO_3.

4.8.2 Fusion mixture, 100 g/l solution

In a suitable beaker, dissolve 25 g of the fusion mixture (4.8.1). Heat if necessary. After cooling, transfer the solution quantitatively into a 250 ml one-mark volumetric flask, dilute to the mark with water and mix well.

4.9 Aluminium, 1 g/l standard solution

Weigh (0,5 ± 0,001) g of aluminium (99,99 % purity) and transfer into a 400 ml beaker. Add 50 ml of hydrochloric acid solution (4.2) and heat gently until aluminium is completely dissolved. After cooling, transfer the solution quantitatively into a 500 ml one-mark volumetric flask, dilute to the mark with water and mix well.

1 ml of this solution contains 1 mg of Al.

4.10 Chromium 1 g/l standard solution

Weigh (0,5 ± 0,001) g of chromium (99,99 % purity) and transfer into a 250 ml beaker. Add 40 ml of hydrochloric acid (4.1) and heat gently until chromium is completely dissolved. After cooling, transfer the solution quantitatively into a 500 ml one-mark volumetric flask, dilute to the mark with water and mix well.

1 ml of this solution contains 1 mg of Cr.

4.11 Cobalt 1 g/l standard solution

Weigh (0,5 ± 0,001) g of cobalt (99,99 % purity) and transfer into a 250 ml beaker. Dissolve it in 5 ml of hydrochloric acid (4.1) and 5 ml of nitric acid (4.3). Heat gently until the metal is dissolved and then boil until nitrous fumes have been expelled. After cooling, transfer the solution quantitatively into a 500 ml one-mark volumetric flask, dilute to the mark with water and mix well.

1 ml of this solution contains 1 mg of Co.

4.12 Copper 1 g/l standard solution

Weigh (0,5 ± 0,001) g of copper (99,99 % purity) and transfer into a 250 ml beaker. Dissolve it in 10 ml of nitric acid solution (4.4). Heat gently until the metal is dissolved, then boil until nitrous fumes have been expelled. After cooling, transfer the solution quantitatively into a 500 ml one-mark volumetric flask, dilute to the mark with water and mix well.

1 ml of this solution contains 1 mg of Cu.

4.13 Manganese 1 g/l standard solution

The manganese used to prepare the solution is released from superficial oxide possibly present by introducing a few grams of metal in a 250 ml beaker containing 150 to 160 ml of water and 15 to 20 ml of sulphuric acid (4.6). Shake and after a few seconds, allow the solution to settle and add water. Repeat the water cleaning several times. Remove the metallic manganese and rinse with acetone. Dry the metal in an oven at 100 °C for 2 minutes or with a hair dryer. Cool in a desiccator.

Weigh (0,5 ± 0,001) g of this purified manganese and transfer into a 250 ml beaker. Dissolve it in 5 ml of hydrochloric acid (4.1) and 10 ml of nitric acid solution (4.4). Heat gently until the metal is dissolved. After

cooling, transfer the solution quantitatively into a 500 ml one-mark volumetric flask, dilute to the mark with water and mix well.

1 ml of this solution contains 1 mg of Mn.

4.14 Molybdenum 1 g/l standard solution

Weigh (0,5 ± 0,001) g of molybdenum (99,99 % purity) and transfer into a 250 ml beaker. Dissolve it in 10 ml of hydrochloric acid (4.1) and 10 ml of nitric acid (4.3). Heat gently until the metal is dissolved. After cooling, transfer the solution quantitatively into a 500 ml one-mark volumetric flask, dilute to the mark with water and mix well.

1 ml of this solution contains 1 mg of Mo.

4.15 Nickel 1 g/l standard solution

Weigh (0,5 ± 0,001) g of nickel (99,99 % purity) and transfer into a 250 ml beaker. Dissolve it in 10 ml of nitric acid solution (4.4). Heat gently until the metal is dissolved, then boil until nitrous fumes have been expelled. After cooling, transfer the solution quantitatively into a 500 ml one-mark volumetric flask, dilute to the mark with water and mix well.

1 ml of this solution contains 1 mg of Ni.

4.16 Phosphorus 1 g/l standard solution

Weigh (2,197 ± 0,001) g of dried potassium dihydrogen phosphate, transfer into a 250 ml beaker and dissolve it with water. Transfer the solution quantitatively into a 500 ml one-mark volumetric flask, dilute to the mark with water and mix well.

1 ml of this solution contains 1 mg of P.

4.17 Tin 1 g/l standard solution [freshly prepared]

Weigh (0,5 ± 0,001) g of tin (99,99 % purity) and transfer into a 250 ml beaker. Dissolve it in 50 ml of hydrochloric acid (4.1). Heat gently until the metal is dissolved. After cooling, transfer the solution quantitatively into a 500 ml one-mark volumetric flask, dilute to the mark with water and mix well.

1 ml of this solution contains 1 mg of Sn.

4.18 Vanadium 1 g/l standard solution

Weigh (0,5 ± 0,001) g of vanadium (99,99 % purity) and transfer into a 250 ml beaker. Dissolve it in 30 ml of hydrochloric acid (4.1) and 10 ml of nitric acid (4.3). Heat gently until the metal is dissolved. After cooling, transfer the solution quantitatively into a 500 ml one-mark volumetric flask, dilute to the mark with water and mix well.

1 ml of this solution contains 1 mg of V.

NOTE Standard solutions whose preparations are described under items 4.9 to 4.18 can be replaced by commercial available standard solutions, provided that they are supplied by a recognised producer and their traceability fully documented.

4.19 Internal reference element solution, 1 g/l

Choose a suitable element to be added as internal reference and prepare a 1 g/l solution.

NOTE Elements as Cd, Fe, Sc and Y were used for this purpose during the validation precision test of this method.